

PART 53—FORMS

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13. ABSTRACT (Maximum 200 words) This AASERT grant provided augmentation funds that helped support US citizen graduate student research in the area of surface science. These efforts focussed on gas-surface reactions, collisional energy transfer, and surface metallurgy, with the latter area encompassing such important topics as metallic surface structure, force constants and bonding at interfaces, structural change due to adsorption/absorption, step dynamics, and the kinetics of surface oxidation and corrosion. This award helped accelerate and expand an AFOSR program which has made significant contributions in areas of DoD interest, and which has a demonstrated record of achievement in training US citizens in critical areas of technological need. The students were trained on state-of-the-art scattering instruments and scanning probe microscopes. This program initially dealt with the interaction of neutral particle, electron, and optical beams with well-characterized surfaces. New activities involving scanning tunneling/atomic force microscopies in air, electrochemical, and UHV environments came online during the latter part of the grant. These studies were motivated by a desire to understand and control surface reactions, such as surface oxidation, and the related technological need to characterize the growth and physical properties of interfaces, thin films, and adsorbed overlayers.				
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Augmentation Award for Surface Science Research Training**AFOSR-F49620-93-1-0536****Final Technical Report for September 1, 1993 - August 31, 1996****Submitted by: Steven J. Sibener****Department of Chemistry and The James Franck Institute
The University of Chicago, 5640 South Ellis Avenue
Chicago, Illinois 60637****Contents**

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2. Introduction

This AASERT grant provided augmentation funds that helped support US citizen graduate student research in the area of surface science as it pertains to gas-surface reactions, collisional energy transfer, and surface metallurgy, with the latter area encompassing such important topics as metallic surface structure, force constants and bonding at interfaces, force constant and structural change due to adsorption/absorption, step dynamics, and the kinetics of surface oxidation and corrosion. This augmentation award helped accelerate and expand an AFOSR program which has made significant contributions in areas of DoD interest, and which has a demonstrated record of achievement in attracting and training US citizens in critical areas of technological need. The graduate (and undergraduate) students that have been able to join our research group are being trained on state-of-the-art scattering instruments that were constructed with substantial DoD support, and have gone on to major research positions in US laboratories. This research program specifically deals with the interaction of neutral particle, electron, and optical beams with well-characterized surfaces. New supporting initiatives involving scanning tunneling and atomic force microscopies in air, electrochemical, and UHV environments are also getting underway. These studies are motivated by a desire to understand and control surface reactions, such as surface oxidation, and the related technological need to characterize the growth and physical properties of interfaces, thin films, and adsorbed overlayers.

The AASERT supported students participated in research projects that were primarily funded by our "core" AFOSR sponsored program, under the auspices of AFOSR-F49620-93-1-0044, "Dynamics of Gas-Surface Interactions". Some additional benefit has also been realized in our corrosion program, funded under AFOSR-F49620-93-1-0423, "Scanning Tunneling Microscopy Studies of the Morphology and Kinetic Pathways for Corrosion Reactions of Stressed Materials. The presence of these AASERT supported students certainly led to an acceleration and broadening of the work in our electron scattering, neutral particle scattering, and scanning probe microscopy/electrochemistry laboratories.

3. Impact of AASERT Funding

The primary goal of this research grant was to augment research activities in our electron scattering, neutral particle scattering, and new STM/AFM/Electrochemistry facilities. This goal was clearly achieved: this AASERT grant has helped partially support several US citizen graduate students in the areas of surface chemistry, electrochemistry, metallic oxidation, and materials science over the past three years (see Personnel section for details). The presence of some of these students in the PI's research group would not have been possible without this grant. AFOSR funded research programs were significantly accelerated and enhanced by the presence of these individuals, with their efforts focussing on such important topics as metallic oxidation and corrosion kinetics, interfacial electrochemistry, and the surface structure and stability of metallic interfaces during the initial stages of metallic oxidation. The first of the AASERT students supported by this grant, Mike Stirniman, has now graduated, taking a postdoctoral position at Battelle Pacific Northwest National Laboratory. Tom Pearl, Dan Gaspar, Ben Zion, and John Mueller directly benefitted from this grant after Mike's departure. Their accomplishments are described in Section 5 of this document.

4. Personnel Supported and Associated with This Program

Students who received partial or full support from this AASERT grant:

Mike Stirniman (now at Battelle Pacific NW National Laboratory; Ph.D. March 1995 entitled: "The Interaction of Oxidizing Adsorbates with the Ni(111) Surface")

Daniel J. Gaspar

Tom Pearl

Ben Zion

John Mueller

N.B. Mike and Ben worked on Ni oxidation, Dan on oxygen induced step doubling and step phonon measurements, while Tom and John worked with scanning probe microscopy.

Others who benefitted from their work (not AASERT supported) include:

Steven J. Sibener, Professor and PI

Kevin Gibson, Research Scientist

Wei Li, Postdoctoral Fellow

Errol Sanchez, Graduate Student

Wenhai Liu, Graduate Student

Jongin Hahn, Graduate Student

Bill Isa, Undergraduate

5. Accomplishments/New Findings

During the first year, 1994-95, this AASERT grant helped support two US citizen graduate students in the area of surface chemistry and materials science. These students were **Daniel J. Gaspar** and **John Mueller**. Dan primarily conducted his research in our high resolution neutral particle surface scattering laboratory. He was concerned with the vibrational properties of stepped metallic surfaces, and how these surfaces were modified during various stages of oxidation. He also helped establish our new scanning tunneling and atomic force microscopy facility. In particular, he designed an anechoic chamber for minimizing acoustical noise in our air and electrochemistry imaging experiments. John was a new student who spent the summer quarter becoming familiar with our new endeavor in stress effects in surface chemistry. Preliminary images were obtained of oxidized aluminum surfaces without the presence of an externally applied stress field. John has since left the U of C doctoral program and is now at Vanderbilt.

During the second year of this grant the AASERT students were **Daniel J. Gaspar**, **Tom Pearl**, **Mike Stirniman**, and **Ben Zion**. Dan continued his research in our high resolution neutral particle surface scattering laboratory. Tom began designing our new UHV-STM facility. Mike was a superb graduate student in our combined molecular beam/electron scattering facility. His

work on synergistic effects involving electrons during metallic oxidation has received widespread attention. Mike graduated in March 1995 and is now a postdoctoral fellow at Battelle Pacific Northwest National Laboratory. Finally, Ben was then a new graduate student continuing and extending the metallic oxidation experiments initiated during Mike's doctoral research.

Mike Stirniman's Ph.D. thesis (awarded March 1995) contains major findings dealing with the oxidation/corrosion kinetics of Ni(111). In particular, this work demonstrated that low fluxes of electrons can create nucleation centers which significantly accelerate the oxidation of this interface. These findings have overturned the literature on this topic. Moreover, his work (with postdoctoral fellow Wei Li) delineated the correct oxidation behavior for Ni(111) both with and without the presence of electrons. The synergistic effects on metallic oxidation induced by electrons were quantified; cross-sections for this phenomenon are given in several papers. Finally, kinetic models were derived which globally fit the oxidation data, whether electrons are present or not, and, if present, for electrons spanning the range from about 30 eV-2keV with fluxes from microamps through milliamps. *In situ* vibrational spectroscopy (HREELS) was also used to follow the transition from the initially adsorbed chemisorbed oxygen to the formation of bulk metallic oxide. As stated above, Ben Zion is continuing this work.

Major discoveries were also made by Licheng Niu and AASERT fellow Dan Gaspar, who successfully mapped out the surface vibrational properties of a stepped metallic surface, Ni(977). These results, initially presented in a *Science* article in May 1995, are the first in-depth measurements of step-induced phonons on a crystalline surface. Moreover, analysis reveals significant anisotropy in the force field near the step edge, with all forces being substantially smaller than in the bulk. Such measurements give valuable information on metallic bonding and interface stability near extended surface defects. Our findings have also shown that the forces in the (111) terraces of the stepped interface differ substantially from those of a smooth (i.e., non-stepped) Ni(111) surface. New and very powerful scattering methodologies were also introduced in these studies which will be of general future use in studying the surface forcefields of clean and adsorbate covered surfaces. These measurements were carried out in our high resolution helium scattering instrument, which you can view as a surface-sensitive analog to inelastic neutron scattering. Our current energy resolution is on the order of 1 cm⁻¹. Wenhai Liu and AASERT fellow Dan Gaspar continued this work.

Finally, Licheng and Dan also examined the detailed kinetics of the initial stages of oxidation for a stepped metallic surface. The focus of this project was the remarkable step-doubling and subsequent step-singling (undoubling) which is induced by adsorbed oxygen. Here the surface is transformed from one having 8 atom wide terraces and 1 atom high steps to one which has 15 atom wide terraces and 2 atom high steps. This transformation is driven by the adsorption of oxygen at the step edges. These experiments examined the detailed kinetic mechanisms and energetics of these transformations, as well as the stability regimes for these reconstructions. We mapped out the structural changes which occur on a stepped, i.e., imperfect, metallic interface upon exposure to gas phase O₂. These include step doubling, step singling, the formation of two different ordered oxygen overlayers, and, finally, the onset of bulk oxide formation. These results delineate the sequence of mechanistic steps which occur during the initial stages of oxidation of a stepped metallic interface which precede the onset of bulk oxidation, findings which are important for developing an improved understanding of metallic oxidation and corrosion. This work has attracted wide-spread attention, with several theory groups going after the electronic, structural, and kinetics aspects of our work.

Finally, during the third year of this grant, AASERT fellow Tom Pearl designed the layout for our new STM laboratory as well as our new ultra-high vacuum STM apparatus. The lab renovation (funds supplied by the Univ. of Chicago) cost approximately \$100K. and was recently completed to our specifications. This new laboratory currently holds a new UHV-STM instrument as well as our new air/electrochemical STM/AFM. In addition to the UHV-STM, the

new air-levitated vacuum chamber houses instrumentation for low energy electron diffraction (LEED), Auger spectroscopy, thermal desorption, and has provisions for XPS and UPS photoelectron spectroscopies as well as other optical spectroscopies. The system is based on a modified "micro-STM" from Omicron Corporation. This microscope has already generated superb images in air, and we anxiously await its inauguration in a vacuum environment.

When the instrument becomes operational, it will provide a superb real-space complement to our atomic and electron scattering labs. Tom Pearl will begin with experiments on stepped metallic surfaces, expanding our prior work on step-localized surface vibrations and interface restructuring during the initial stages of metallic oxidation. He will in particular use the STM to quantify step structure and atom mobility along step-edges during interface oxidation. These real-space imaging measurements will help us to further refine our current understanding of the sequence of structural changes which occur on a stepped, i.e., imperfect, metallic interface upon exposure to gas phase O_2 . These include step doubling, step singling, the formation of two different ordered oxygen overlayers, and, finally, the onset of bulk oxide formation. This work has attracted wide-spread attention, with several theory groups going after the electronic, structural, and kinetics aspects of our work. Work on metallic alloys will also be part of this new STM program. The reader is referred to recent publications from our group for more background material on this topic:

Observation of Phonons Localized at Step Edges: A Route to Understanding Forces at Extended Surface Defects, L. Niu, D.J. Gaspar, and S.J. Sibener, *Science* **268**, 847 (1995).

Vibrational Dynamics of a Stepped Metallic Surface: Step-Edge Phonons and Terrace Softening on Ni(977), L. Niu, D.D. Koleske, D.J. Gaspar, and S.J. Sibener, *J. Chemical Physics* **102**, 9077 (1995).

Reconstruction Kinetics of a Stepped Metallic Surface: Step Doubling and Singling of Ni(977) Induced by Low Oxygen Coverage, L. Niu, D.D. Koleske, D.J. Gaspar, S.F. King and S.J. Sibener, *Surface Science* **356**, 144 (1996).

Tom Pearl will also use the STM as a spatially localized source of electrons, attempting to elucidate further information on how electrons synergistically enhance metallic oxidation. This will encompass both nucleation issues as well as the actual electron attachment process. These experiments will extend our work on electron stimulated oxidation, experiments which have received widespread attention in both the metallic oxidation and semiconductor patterning communities. The reader is referred to recent work from our group for more details:

Electron Stimulated Oxidation of Ni(111) at Low Temperature, Wei Li, M.J. Stirmiman, and S.J. Sibener, *Surface Science Letters* **322**, L593 (1995).

Inelastic Electron Scattering Study of Metallic Oxidation: Synergistic Effects Involving Electrons During the Low Temperature Oxidation of Ni(111), Wei Li, M.J. Stirmiman, and S.J. Sibener, *J. Vacuum Science and Technology A*, **13**, 1574 (1995).

Electron Stimulated Oxidation of the Ni(111) Surface: Dependence on Substrate Temperature and Incident Electron Energy, M.J. Stirmiman, Wei Li, and S.J. Sibener, *J. Chemical Physics* **103**, 451 (1995).

Finally, in addition to Tom Pearl, two other students directly benefitted from this AASERT grant during the third grant year. These were **Dan Gaspar** and **Ben Zion**, whose activities were previously described. Dan's focus moved to the energetics of kink formation on stepped surfaces, while Ben continued his efforts on Ni oxidation.

During the next grant period Ben (and another new student, **Julie Jaasma**) will construct an RF plasma beam source which will allow us to expand our studies of surface oxidation to include highly energetic oxidants. This atomic oxygen beam source will be capable of generating intense beams of excited oxidants, such as $O(^3P)$, $O(^1D)$, and $O_2(^1\Delta)$. Such a plasma beam source was originally developed by the PI for work in gas phase kinetics, and has been successfully duplicated by other groups over the past decade. Modern broad-band RF electronics should allow it to run even more efficiently than in the past. It will be a superb addition to our studies of surface oxidation kinetics.

Further details of the research accomplishments of the AASERT students can be found in the various reports filed for AFOSR-F49620-93-1-0044, "Dynamics of Gas-Surface Interactions" and AFOSR F49620-93-1-0423, "Scanning Tunneling Microscopy Studies of the Morphology and Kinetic Pathways for Corrosion Reactions of Stressed Materials. Work is continuing under the auspices of our new core grant F49620-96-1-0084 "Surface Chemistry, Vibrational Dynamics, and Structural Stability of Metallic Interfaces".

6. Publications

Papers published that involved AASERT supported students were:

Interadsorbate Interactions in the $c(4 \times 2)$ NO/Ni(111) System, M.J. Stirniman, Wei Li, and S.J. Sibener, J. Chemical Physics 102, 4699 (1995).

Electron Stimulated Oxidation of Ni(111) at Low Temperature, Wei Li, M.J. Stirniman, and S.J. Sibener, Surface Science Letters 329, L593 (1995).

Inelastic Electron Scattering Study of Metallic Oxidation: Synergistic Effects Involving Electrons During the Low Temperature Oxidation of Ni(111), Wei Li, M.J. Stirniman, and S.J. Sibener, J. Vacuum Science and Technology A, 13, 1574 (1995).

Electron Stimulated Oxidation of the Ni(111) Surface: Dependence on Substrate Temperature and Incident Electron Energy, M.J. Stirniman, Wei Li, and S.J. Sibener, J. Chemical Physics 103, 451 (1995).

Observation of Phonons Localized at Step Edges: A Route to Understanding Forces at Extended Surface Defects, L. Niu, D.J. Gaspar, and S.J. Sibener, Science 268, 847 (1995).

Vibrational Dynamics of a Stepped Metallic Surface: Step-Edge Phonons and Terrace Softening on Ni(977), L. Niu, D.D. Koleske, D.J. Gaspar, and S.J. Sibener, J. Chemical Physics 102, 9077 (1995).

Reconstruction Kinetics of a Stepped Metallic Surface: Step Doubling and Singling of Ni(977) Induced by Low Oxygen Coverage, L. Niu, D.D. Koleske, D.J. Gaspar, S.F. King and S.J. Sibener, Surface Science 356, 144 (1996).

7. Interactions with DoD/Transitions

7a. Connections with DoD

The PI and his group have attended several DoD meetings on the topic of corrosion and aging aircraft. For example:

- Graduate student Mike Stirniman participated in the "Joint AFOSR-AFMC/EN Aging Aircraft Conference" [Workshop on Aging Aircraft Research] that was held from 27-28 April 1993 at Georgia Tech in Atlanta, GA.

- Professor Sibener was an invited participant at the "Second Air Force Aging Aircraft Conference" that was held from 17 May to 19 May at the Oklahoma City Air Logistics Center, Tinker AFB, OK. His talk was entitled "The Initial Stages of Metallic Oxidation", and stimulated much discussion with the participants and sponsors.

- Professor Sibener was also an invited speaker at the AFOSR/URI Meeting on "Corrosion, Tribology, Lubrication, and Materials Fatigue Under Extreme Conditions" that was held from 17-18 August 1994 at the Beckman Institute at the University of Illinois/Urbana-Champaign. His presentation was entitled "The Initial Stages of Metallic Oxidation", and was presented at a much higher technical level than the overview talk presented earlier at Tinker AFB.

- As our electrochemical etching work advances, additional contacts with Tinker AFB and Wright-Patterson are envisioned.

7b. DoD Consulting of the PI

The PI was a charter member of the Defense Science Study Group of the Institute for Defense Analyses. He is currently a consultant for IDA.

7c. Transitions

1. Title: Electronics for high-impedance STM imaging
Purpose: Circuitry needed for STM imaging of molecular overlayers
Recipient: Mark Greenbaum, Topometrix Corporation (708 717 0566)
2. Title: Ultra-high vacuum technology for atomic traps
Purpose: Generation of ultra-low temperature atomic beams for atomic/synchrotron physics and small-scale pattern deposition
Recipient: Bruce Zabransky, Argonne National Lab (630-252-4046)
3. Title: Multiple supersonic molecular beam methods of materials growth
Purpose: Improved growth of advanced semiconductors
Recipient: Dr. Hiroshi Kajiyama/Dr. Hrvoje Petek
Advanced Research Laboratory, Hitachi Ltd., Japan
4. Title: Electron enhanced oxidation of materials
Purpose: Improved oxidation and patterning of semiconductors
Recipient: Dr. Wilson Li, Intel Corp., 408-765-2837

8. Biographical Information of the PI: STEVEN J. SIBENER

August 1996

Born

April 3, 1954; Brooklyn, New York

Education

1971-1975 University of Rochester, Rochester, New York.
Sc.B. in Chemistry awarded with High Distinction, 1975.
B.A. in Physics awarded with Distinction, 1975.

1975-1979 University of California, Berkeley.
M.S. in Chemistry, 1977.
Ph.D. in Chemistry, 1979.
Research with Professor Yuan T. Lee.

Professional Experience

Eastman Kodak Research Laboratories, Photographic Research Division, Summer 1974: Electrostatic properties of polymers and polymer-metal interfaces.

Eastman Kodak Research Laboratories, Physics Division, Solid State Physics Research Laboratory, Summer 1975: Oxide growth on GaAsP for MOS fabrication.

Bell Laboratories Postdoctoral Fellow, September 1979 - August 1980. Research with Dr. M.J. Cardillo involving molecular beam scattering from single crystal surfaces.

The University of Chicago, The James Franck Institute and The Department of Chemistry:
Assistant Professor, August 1980 - June 1985.
Associate Professor, July 1985 - June 1989.
Professor of Chemistry, July 1989 - .

Honors and Awards

Chairman, Div. of Chemical Physics, American Physical Society, 1996.
Visiting Fellow, Joint Institute for Laboratory Astrophysics, University of Colorado, Boulder, 1992-93.
Marlow Medal of the Faraday Division of the Royal Society of Chemistry, 1988.
IBM Faculty Development Award, 1984-86.
Alfred P. Sloan Foundation Research Fellow, 1983-87.
Camille and Henry Dreyfus Young Faculty in Chemistry Award, 1980.
Gulf Oil Research Fellow, University of California, Berkeley.
American Institute of Chemists Award, University of Rochester.
American Chemical Soc. Div. of Colloid and Surface Chemistry Undergraduate Thesis Competition Honorable Mention: "The Shape of Liquid Interfaces," 1975.
Regional Scholar for New York City, University of Rochester.

Associations

Phi Beta Kappa, American Physical Society,
American Chemical Society, Royal Society of Chemistry,
Sigma Xi, AAAS, American Vacuum Society

S.J. Sibener**Curriculum Vitae (Continued)****Invited Lectureships**

Physikalisches Institut der Universität Erlangen-Nürnberg, 1988
Chemical Physics Institute Retreat Speaker, U of Oregon at Eugene, 1992.
ACS Lecturer of the Analytical Chemistry Division, University of Wisconsin, Madison, 1992.

Consulting

Dow Chemical USA (1982-1985)
Teltech Resource Network (1985-)
Institute for Defense Analyses (1985)

Professional Activities

Vice-Chairman, 1985 Gordon Research Conference on the Dynamics of Gas-Surface Interactions
Member, Defense Science Study Group--Institute for Defense Analyses (1985 - 1988).
Member, Materials Research Laboratory Policy Committee, The University of Chicago (1987 -).
Chairman, 1987 Gordon Conference on the Dynamics of Gas Surface Interactions.
Member, International Advisory Committee of the Vibrations at Surfaces V Conference (September 1987).
Member, Board of Trustee's Visiting Committee for the College of Arts and Science, University of Rochester (July 1, 1987 - June 30, 1993).
Member, Council of the University Senate, The University of Chicago (September 1989 - September 1992).
Member, IQEC '90 Program Committee (May 1990).
Member, Physical Electronics Conference Advisory Committee (1991).
Member, Physical Electronics Conference Committee (1991-1994).
Organizer, Symposium on "Surface Chemistry from Reaction Dynamics Through Materials Growth", August 1992. ACS National Meeting, Washington, DC.
Co-Chairman, Symposium on "Laser Techniques for Surface Science", January 1994, SPIE/OE LASE '94, Los Angeles, CA.
Chairman, ACS Irving Langmuir Prize Canvassing Committee (1995).
Division of Chemical Physics, American Physical Society:
Vice-Chair (1994), Chair-Elect (1995), Chair (1996).

Research Interests

Surface Chemistry & Physics; Interfacial Kinetic Processes; Chemical Physics; Reaction Dynamics; Electrochemistry; Materials Growth and Thin Film Dynamics; Molecular Beam Scattering; Surface Metallurgy; interface oxidation, vibrational dynamics, alloys, phase transitions; Nanostructures; STM/AFM studies of surface phenomena.

DoD Consulting of the PI

The PI was a charter member of the Defense Science Study Group of the Institute for Defense Analyses. He is currently a consultant for IDA.

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